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Aminomethylation of Thioureas with *N*,*N*-Dimethyl-1-(triethylsiloxy)methanamine, Involving Amino Group Exchange[1]

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Abstract—The *O*-triethylsilylated hemiaminal $Et_3SiOCH_2NMe_2$ readily transfers the Me_2NCH_2-group to various thioureas under mild conditions and without catalysts or co-reagents. In the reaction with PhNHC(=S)·NHPh, the initially formed mono-substituted derivative $PhNHC(=S)NPhCH_2NMe_2$ readily rearranges to produce the unsymmetrical thiourea $PhNHC(=S)NMe_2$ and hexahydro-1,3,5-triphenyl-1,3,5-triazine.

Keywords: *O*-silylated hemiaminal, aminomethylation, thioureas, thiourea rearrangements

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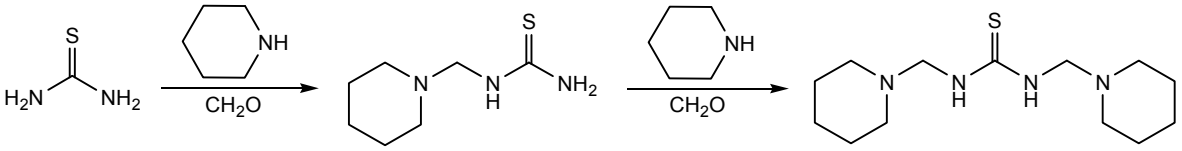
Thioureas have long been studied as useful synthons and bioactive materials [1], and their aminomethylation reactions have received attention [2]. For example, the reaction of the parent thiourea, $H_2N(C=S)NH_2$, with paraformaldehyde and piperidine has been shown to lead to both mono- and di-substituted materials, as illustrated in reaction (1) (Scheme 1) [2a].

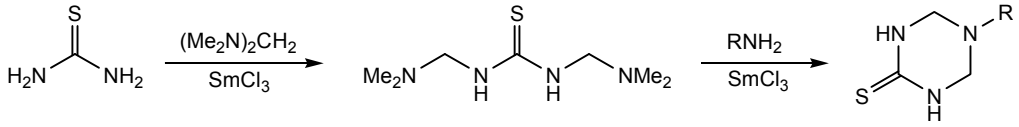
The well-known Mannich reagent *N*,*N*,*N*',*N*'-tetramethyldiaminomethane ($Me_2NCH_2NMe_2$) has also been shown to react with thiourea to form a similar bis-Me_2NCH_2-substituted product that is a useful reagent for further reaction with primary amines to form 1,3,5-triazinane-2-thiones [2]. However, this reagent needs the presence of metal catalysts, e.g. Cu or Sm, reaction (2) (Scheme 2) [2b].

Scheme 1.



(1)

Scheme 2.



(2)

N,*N*-Dimethyl-1-trimethylsiloxymethanamine $Me_3SiOCH_2NMe_2$ was first synthesized in 1981 by the Mironov's group [3] by the reaction of Me_2NSiMe_3 with formaldehyde. The authors demonstrated its reactions with halosilanes R_3SiX to form Me_2NCH_2X, with excess *N*,*N*-dimethyl-1-trimethylsilylmethan-amine to form diamines [reaction (3a)], and with hydroaminosilane $BuNHSiMe_3$ to form hexahydro-1,3,5-tributyl-1,3,5-triazine [reaction (3b)] [4]

[1] This article is dedicated to many excellent Russian exponents and proponents of organosilicon chemistry, past, present, and future.

$$Me_3SiOCH_2NMe_2 + Me_3SiX \rightarrow Me_2NCH_2NMe_2 + Me_3SiOSiMe_3 \quad (3a)$$

$$3\ Me_3SiOCH_2NMe_2 + 3\ Me_3SiNHBu \rightarrow (BuNCH_2)_3 + 1.5\ Me_3SiOSiMe_3 \quad (3b)$$

We earlier reported the utility of *N*,*N*-dimethyl-1-triethylsiloxymethanamine $Et_3SiOCH_2NMe_2$ (**1**) (an *O*-silylated hemiaminal) as a versatile and very efficient aminomethylating reagent that requires neither catalysts nor high temperatures [5–7]. Thus, compound **1** readily reacts with a range of EH, E = O, S, N, materials to form the corresponding E–CH_2NMe_2 products in high yield. For the corresponding E = P chemistry, catalysts are required for efficient chemistry.

We now report the utility of **1** for aminomethylation of thioureas **2**, **3**, and **4**, that results in new previously unknown materials and surprising chemistry (Scheme 3).

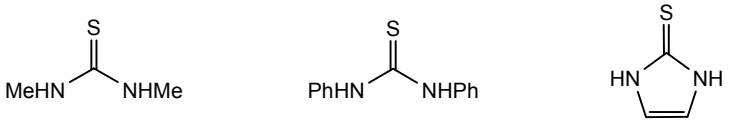
The reactions of compound **1** were studied in dichloromethane without any catalytic reagent and were fast and completed in a few hours at room temperature as determined by ^{13}C and ^{1}H NMR monitoring. The reactions of both *N*,*N*-dimethylthiourea (**2**) and 1,3-dihydroimidazole-2-thione (**4**) were unremarkable and led initially to monosubstitution in both cases to form compounds **5** and **6**, respectively, reactions (4) and (5) (Scheme 4). The reaction of imidazole **4** continued in the presence of excess **1** to form disubstituted product **7** (both **6** and **7** are new compounds). Contrary to the chemistry of **4**, we never observed a second substitution in the case of thiourea **2**, regardless of the stoichiometry of the reaction. Presumably, the restricted geometry of the NH bonds in **4** accounts for this extra reactivity, although precisely how is outside the scope of our present study.

The single crystal structure of 1,3-bis(*N*,*N*-dimethylaminomethyl]-1,3-dihydro-2*H*-imidazole-2-thione (**7**) is presented in Fig. 1. The molecule has a C^2 symmetry through the C=S bond, and the bond lengths and angles are as expected. The crystal structure is stabilized by hydrogen bonds (HBs) between N^2 and H^{4B} (2.850 Å) forming R_2^2 (6) motifs, and generating chains linked by this motif (Fig. 2). The 3-D architecture is generated by the motif C_2^1 (12) formed by the bidentate interaction of S^1 with two symmetrically equivalent H^{4A} (2.876 Å).

The reaction between **1** and *N*,*N*-diphenylthiourea (**3**) was more interesting and produced new chemistry as opposed to simply new products. By ^{1}H and ^{13}C NMR monitoring we could observe the rapid formation of a mono-aminomethylation product, PhNH(C=S)PhNCH_2NMe_2 (**8**). However, all attempts to isolate this material resulted in the recovery of two major products, the asymmetrical thiourea PhNH(C=S)·NMe_2 (**9**) [8], and hexahydro-1,3,5-triphenyl-1,3,5-triazine (**10**) (Scheme 5). This chemistry represents a new molecular rearrangement for such a structural unit.

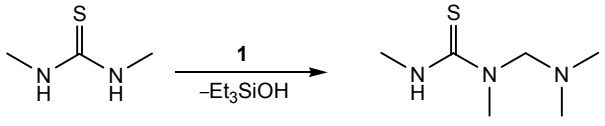
If the initial reaction between compounds **1** and **3** is monitored after the reaction to form **8** has gone to completion, it can be seen that **8** is slowly converted to **9** and **10**, even at room temperature. However, if the

Scheme 3.



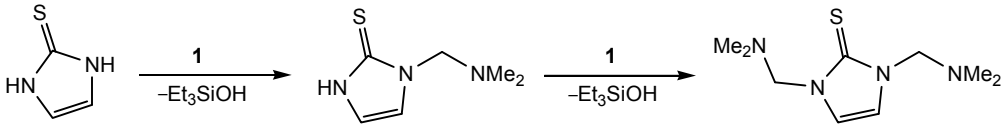
2 **3** **4**

Scheme 4.



(4)

2 **5**



(5)

4 **6** **7**

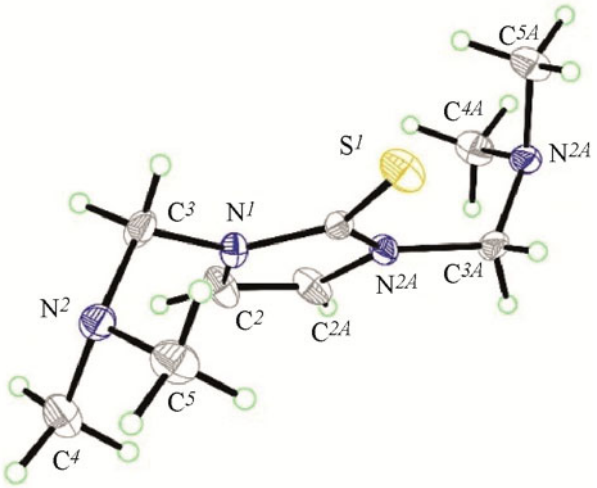


Fig. 1. Thermal ellipsoid rendering of molecule **7** (the thermal ellipsoids are drawn at a 50% probability level and hydrogens are drawn as spheres of arbitrary radii).

initially formed solution of **8** is heated to 60°C, the transformation is complete within 40 min.

A plausible mechanism associated with this transformation, involving an intramolecular base substitution at the thiocarbonyl group by the strongly basic Me_2N functionality, is outlined in Scheme 6.

Attempted isolation of triamine **8** using many solvent systems to recrystallize the solid gave a product that spectroscopically appeared to be pure. Sometimes from such attempts, in addition to **9** and **10**, we isolated small amounts of a new aminomethanol, $PhHN(C{=}S)NPhCH_2OH$ (**11**), and could obtain a crystal structure of this unusual material (Fig. 3).

In the solid state, compound **11** forms an intramolecular HB with the sulfur atom as acceptor (2.629 Å), while forming chains through the O^1-H···N^1 HBs (2.209 Å). At the same time, the C–N and C=S bonds show no significant differentiation in comparison to **7**, suggesting that the electronic factors are similar. The presence of the phenyl group reduces the conjugation of the nitrogen lone pair with the C–S system, as shown by a comparison of the structure in focus with that of the related compound $[CH_3HN(C{=}S)\cdot NHCH_2OH]$ (**12**) [9]. The C–S bond distance of 1.728(8) Å in **12** is considerably reduced compared to 1.6870(11) and 1.678(3) Å for **7** and **11**, respectively. Furthermore, in the case of **12** the intra-molecular HB is absent, indicating that it is probably the result of steric interactions due to the phenyl rings in compound **11**.

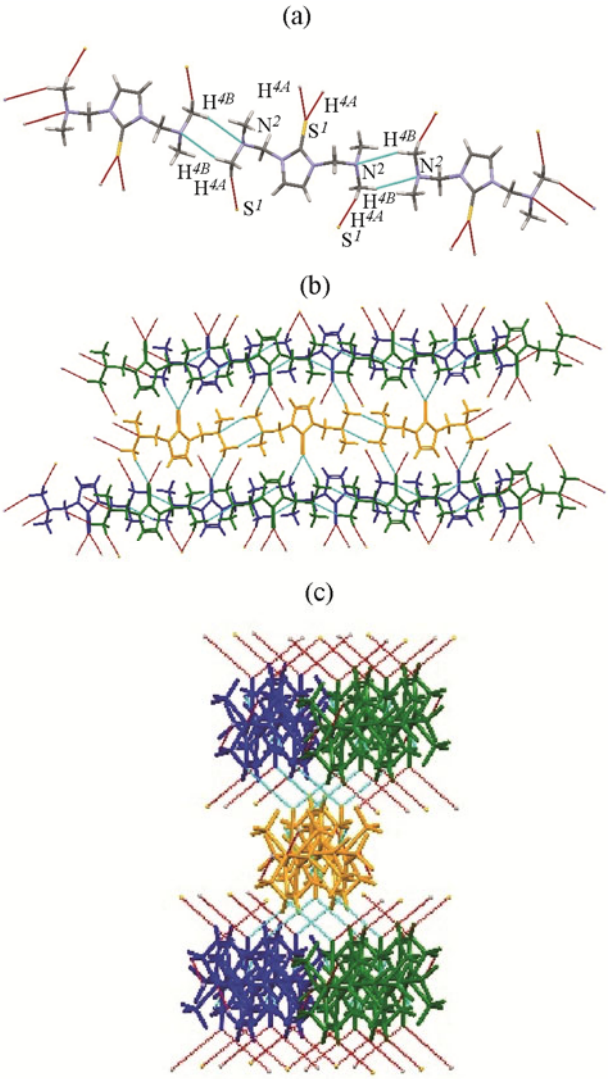


Fig. 2. Views of (a) the chain formed via intermolecular CH···N and CH···S HBs and (b) crystal packing of compound **7**, showing the central HB chain formed by CH···N HBs and its interaction with neighboring chains through CH···S HBs, and (c) lateral view of the same crystal packing.

We surmised that **11** was formed by the hydrolysis of **8** by residual water in our dried solvent systems and thus attempted a treatment of **8** with water. This experiment resulted in no identifiable products.

In another attempt to derivatize **8** we treated it with MeI hoping to obtain a quaternary ammonium salt. This reaction led only to the isolation of methyl *N*,*N*'-diphenylcarbamimidothioate (**13**). After several attempts to get suitable crystals, an acceptable structure was solved in terms of R_1 (Table 1), although with a high R_1 descriptor (Fig. 4). The structure data collected at 100 K showed 4 symmetrically independent mole-

Table 1. Principal crystallographic parameters of compounds **7**, **11**, and **12**

Parameter	**7**	**11**	**12**
Formula	$C_9H_{18}N_4S$	$C_{20}HN_2OS$	$C_{14}H_{14}N_2S$
MW	214.33	317.29	242.33
Crystal system	Monoclinic	Orthorhombic	Monoclinic
Space group	*C2/c*	*Pca2₁*	*P2₁/c*
Z	4	4	16
a, Å	8.2405(2)	18.932(3)	8.6216(6)
b, Å	11.6742(3)	5.5591(8)	15.5092(11)
c, Å	11.8213(3)	12.4216(18)	38.094(3)
α, deg	90	90	90
β, deg	95.6500(10)	90	93.820
γ, deg	90	90	90
V, Å^3	1131.70(5)	1307.3(3)	5082.39
Reflections used	13541	14194	8701
R_{int}	0.0311	0.0418	0.1214
R_1 [$I > 2\sigma(I)$]	0.0304	0.0414	0.1195
q_{min}, q_{max}, deg	3.04, 30.99	2.15, 28.28	2.79, 25.0
T, K	100(2)	298(2)	100(2)

cules in an asymmetric unit cell for a total of 16 molecules in the unit cell.

Examination of the N^1–C^1 [1.366(9) Å] and N^2–C^1 [1.288(9) Å] distances in compound **13** clearly reveals distinctions between the sp^3-C–N and sp^2-C–N bond lengths, whereas the C_{phenyl}–N bond lengths are equivalent: 1.413(8) and 1.425(9) Å, respectively. These observations apply to all the 4 conformers in the asymmetric unit. With respect to the packing, the NH hydrogen forms HBs with the sp^2-C–N nitrogen of a neighboring molecule, generating a chain of the type ···A···B···A···B···. The unit cell contains two different chains: one formed by conformers 1aa and 2ab [N^2···H^3N–N^3 (2.138 Å), N^4···H^1N–N^1 (2.238 Å)] and the other, by conformers 3ac and 4ad [N^8···H^5N–N^5 (2.199 Å), N^6···H^7N–N^7 (2.261 Å)].

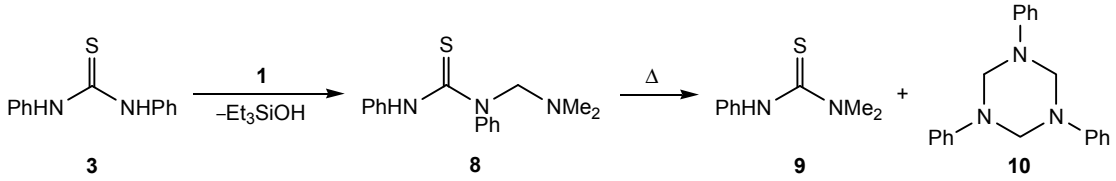
It is of interest to compare the neutral molecule **13** with its HI salt **14**, reported in [10]. The first striking difference is in the conformation adopted by the charged compound (Fig. 4), where the two phenyl groups appear to form a π-stacking motif, although the phenyl rings are divergent rather than parallel. Secondly, the N–C bond distances to the central carbon in salt **14** are statistically equivalent [1.340(6) and 1.309(7) Å], indicating a significant amount of positive charge delocalization involving both N atoms. The values for the N^1–C^1–N^2 angle [122(1)° and 125.5(5)° in **13** and **14**, respectively] and C–S bond length [1.807(15) and 1.750(6) Å] are statistically not differentiable. The N–C_{phenyl} distances in salt **14** are similar to those in the neutral compound and, as might be expected, NH···N HBs are replaced by NH···I interactions.

Overall, the chemistry delineated in this article shows that **1** is an efficient, low energy, Me_2NCH_2-transfer agent to thioureas and requires no catalyst. Additionally, it is clear that a significant new chemistry is to be obtained by further investigation of such simple materials. Scheme 7 illustrates the overall chemistry of compound **8**.

EXPERIMENTAL

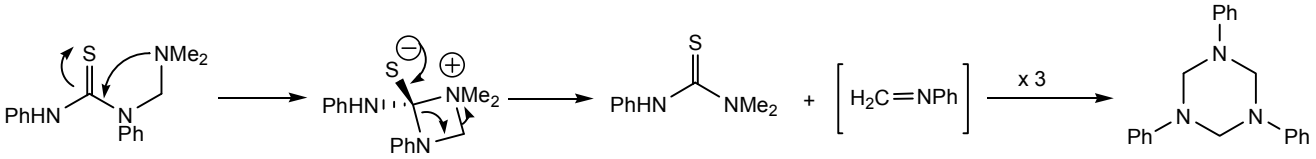
All manipulations were carried out under an argon atmosphere using Schlenk or vacuum line techniques.

Scheme 5. Chemical transformations in the reaction between **1** and PhNH(C=S)NHPh.



3 8 9 10

Scheme 6. Proposed mechanism for transformation of compound **8** to **10**.



8 9 10

THF was distilled under nitrogen from benzophenone ketyl prior to use. Hexane, benzene, and toluene were dried over sodium metal and distilled before use. 1,3-Dimethylthiourea, 1,3-diphenylthiourea, and imidazole-2-thiol were purchased from Sigma-Aldrich. $Et_3SiOCH_2NMe_2$ was synthesized by the reported method [11]. The NMR spectra were recorded on 300 MHz Bruker spectrometer in $CDCl_3$. The SC-XRD analysis of **7** was performed using a Bruker Venture Duo diffractometer with a micro-source and a Photon 200 detector, and the data for **11** were collected on a Bruker APEX CCD diffractometer. Both structures were solved using APEX3 crystallography software suite [12].

Reaction of $Et_3SiOCH_2NMe_2$ with 1,3-dimethylthiourea. In a typical experiment, a 10 mL round-bottom flask was charged with 0.22 g (1.16 mmol) of $Et_3SiOCH_2NMe_2$ and 0.06 g (0.57 mmol) of 1,3-dimethylthiourea in 5 mL dichloromethane. The solution was stirred overnight at room temperature, and the solvent and triethylsilanol were removed under vacuum to obtain 1,2-dimethyl-1-[(dimethylamino)methyl]thiourea $MeNH(C{=}S)NMeCH_2NMe_2$ (**5**) as a colorless oil, yield 90%. The material is thermally stable and does not undergo decomposition even on heating at 70°C for 20 h. ^{1}H NMR spectrum, δ, ppm: 2.18 s (6H, CH_3), 3.03 d (3H, CH_3, *J* 4.8 Hz), 3.32 s (3H, CH_3), 3.75 s (2H, CH_2), 8.21 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 32.2 (Me), 41.4 (Me), 42.1 (Me), 76.4 (CH_2), 186.6 (C=S).

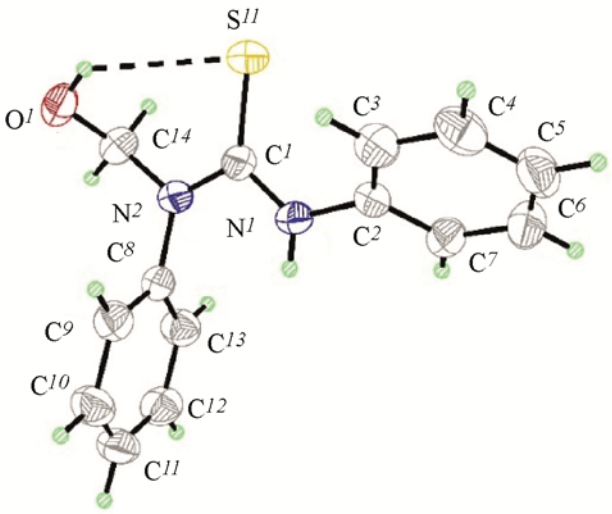


Fig. 3. Thermal ellipsoid rendering of molecule **11** (the thermal ellipsoids are drawn at a 50% probability level and hydrogens are drawn as spheres of arbitrary radii).

Reaction of $Et_3SiOCH_2NMe_2$ with imidazole-2-thiol in a 1 : 1 molar ratio. A 20 mL round-bottom flask was charged with 0.125 g (1.25 mmol) of imidazole-2-thiol and 0.48 g (0.25 mmol) of **1** in 10 mL of dichloromethane. The solution was stirred overnight at room temperature. Volatiles were then removed under vacuum to leave a brown solid, repeated recrystallization of which from a mixture of hexane and dichloromethane gave a 0.7 : 1.0 mixture of 1-(dimethylamino)imidazole-2-thiol (**6**) and 1,3-bis(dimethylamino)imidazole-2-thiol (**7**).

1-(*N*,*N*-Dimethylaminomethyl)-1,3-dihydro-2*H*-imidazole-2-thione (6). ^{1}H NMR spectrum, δ, ppm: 2.33 s (6H, NMe_2), 4.76 s (2H, CH_2), 6.70 d (1H, CH=CH, *J* 3.0 Hz), 6.82 d (1H, CH=CH, *J* 3.0 Hz), 10.59 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 42.5 (NMe_2), 68.0 (CH_2), 113.86, 118.1 (C=C), 161.4 (C=S).

1,3-Bis(*N*,*N*-dimethylaminomethyl)-1,3-dihydro-2*H*-imidazole-2-thione (7). ^{1}H NMR spectrum, δ, ppm: 2.37 s (6H, NMe_2), 4.81 s (2H, CH_2), 6.83 s (2H,

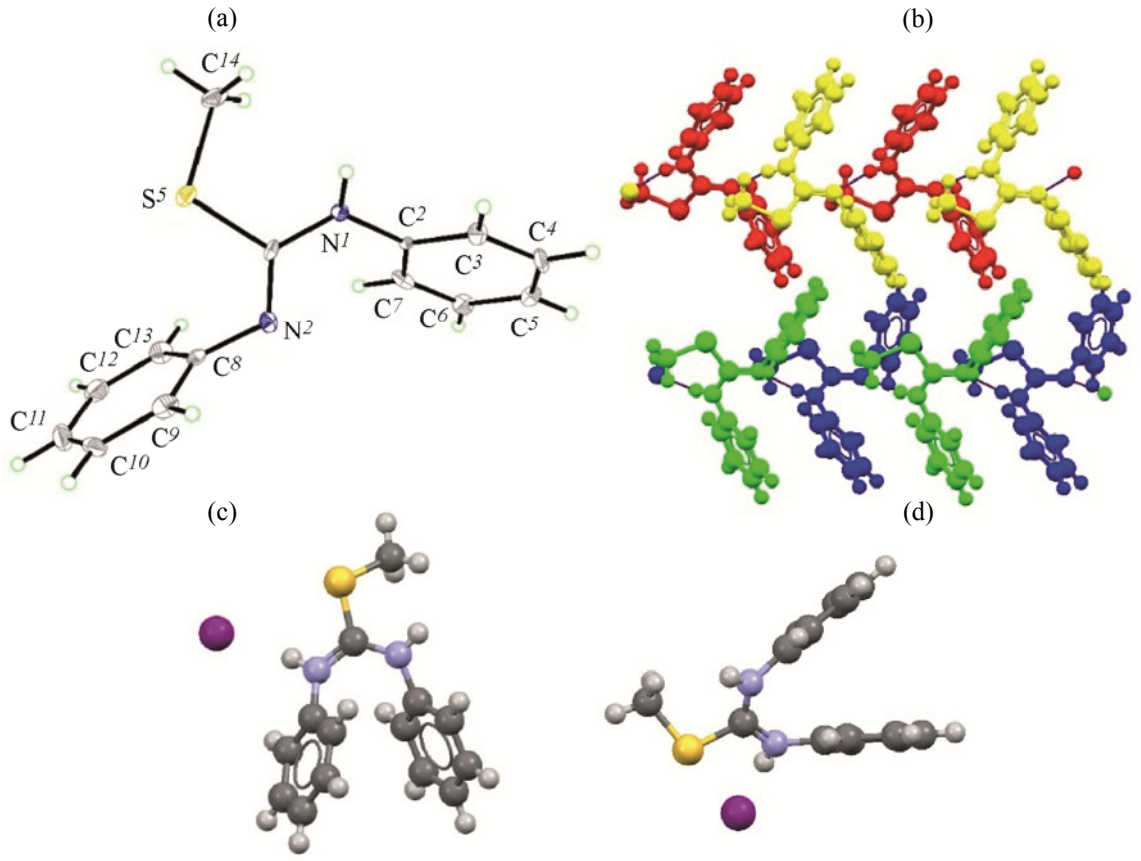


Fig. 4. (a) Ortep view of molecule **13** at a 50% of probability level (shown is just one of the 4 structures in the asymmetric unit cell) and (b) chains formed by interaction of the four conformers: (blue) conformer 1aa, (green) conformer 2ab, (yellow) conformer 3ac, and (red) conformer 4ad. (c) Structure of HI salt **14** [6] and (d) side view of the HI salt.

CH=CH). ^{13}C NMR spectrum, δ, ppm: 42.5 (NMe_2), 68.8 (CH_2), 116.9 (C=C), 163.7 (C=S). Found, %: C 48.75; H 8.13. Calculated, %: C 50.44; H 8.46.

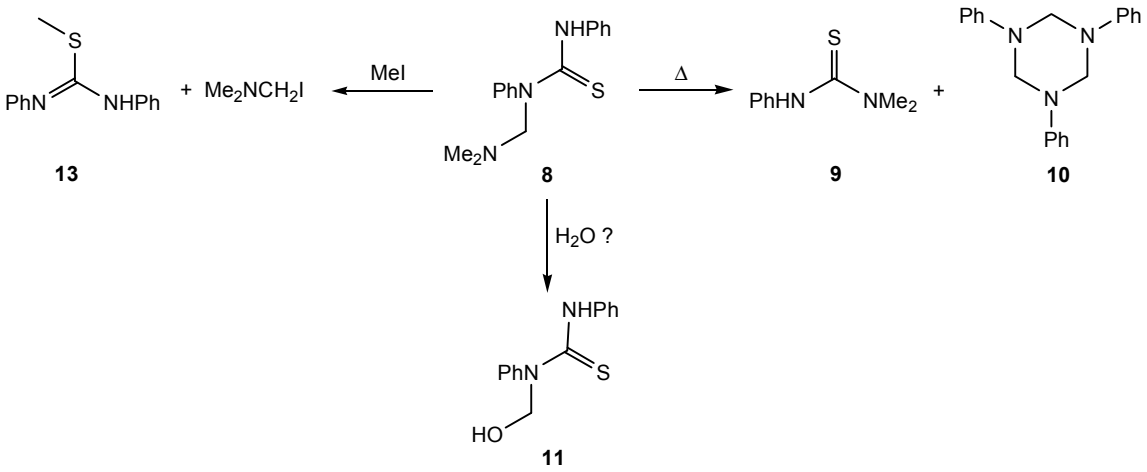
Repeating the reaction of **1** with imidazole-2-thio in 1 : 2 molar ratio in dichloromethane gave a 1 : 2 mixture of compounds **6** and **7**, but using a larger excess of **1** (4-fold) gave compound **7** as a single product (yield 85%).

Reaction of $Et_3SiOCH_2NMe_2$ with 1,3-diphenylthiourea. A 20 mL round-bottom flask was charged with 0.165 g (0.88 mmol) of $Et_3SiOCH_2NMe_2$ and 0.2 g (0.88 mmol) of 1,3-diphenylthiourea in 10 mL of dichloromethane. The solution was stirred overnight at room temperature, after which the solvent and triethylsilanol were removed under vacuum to obtain 1-[(dimethylamino)methyl]-1,2-diphenylthiourea PhNH(C=S)$NPhCH_2NMe_2$ (**8**), yield 80%. This new material is thermally labile and undergoes slow transformation even at room temperature.

The reaction between **1** and 1,3-diphenyl thiourea in 2 : 1 molar ratio, too, resulted in exclusive formation of **8**; unexpectedly, no disubstituted product was obtained. Due to the limited thermal stability of **8**, we could not perform its CHN analysis. ^{1}H NMR spectrum, δ, ppm: 2.30 s (6H, NMe_2), 4.40 s (2H, CH_2), 7.20–7.41 m (10H), 7.80 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 41.4 (Me), 41.9 (Me), 77.3 (CH_2), 124.6, 125.1, 125.3, 127.5, 128.2, 128.5, 129.5, 139.5 (Ph), 184.7 (C=S).

Thermal rearrangement of 1-[(dimethylamino)-methyl]-1,2-diphenylthiourea (8). A Pyrex NMR tube was charged with 0.183 g (0.64 mmol) of compound **8** in 0.3 mL of $CDCl_3$. The tube was sealed under vacuum and immersed in an oil bath maintained at 70°C. After 5 h, the ^{1}H, ^{29}Si and ^{13}C NMR monitoring showed a quantitative transformation of **8** to 1,1-dimethyl-3-phenylthiourea PhNH(C=S)NMe_2 (**9**) [8] and hexahydro-1,3,5-triphenyltriazine $(PhNCH_2)_3$ (**10**).

Scheme 7. Overall chemistry of **8**.



Formation of *N*-hydroxymethyl-*N*,*N*-diphenylthiourea PhNH(C=S)NPhCH$_2$OH (11). A 0.05 g of thiourea **8** was dissolved in 1 mL of dichloromethane in a screw cap vial and left in the refrigerator for a week. A mixture of thioureas PhNH(C=S)NPhCH$_2$OH (**11**), PhNH(C=S)NHPh, and compound **8** was obtained as white crystals. Mechanical separation permitted isolation of thiourea **11** which was subjected to XRD analysis. ^{1}H NMR spectrum, δ, ppm: 4.88 t (1H, OH, *J* 9.0 Hz), 5.47 d (2H, CH$_2$), 7.28–7.55 m (10H), 7.81 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 78.1 (CH$_2$), 125.4, 125.8, 126.6, 127.4, 128.7, 129.2, 138.2, 141.3 (Ph), 181.9 (C=S).

Reaction of 1-[(dimethylamino)methyl]-1,2-diphenylthiourea (8) with MeI. A 20 mL round-bottom flask was charged with a solution of 0.24 g (1.27 mmol) of **1** and 0.29 g (1.27 mmol) of 1,3-diphenylthiourea in 10 mL of dichloromethane. The solution was stirred overnight at room temperature. Excess of MeI (1 mL) was then added with a syringe to the reaction mixture. After 15 min of stirring, a white solid started to precipitate, and the reaction mixture was further stirred for 2 h. The solvent and triethylsilanol were removed under vacuum. The white solid was treated twice with 15 mL of hexane, the extract was filtered, and the solvent was removed to obtain methyl *N*,*N*-diphenylcarbamimidothioate PhN=C(SMe)NHPh (**13**), yield 62% yield, mp 106°C (107°C [13]).

Reaction of Et$_3$SiOCH$_2$NMe$_2$ with 1,3-dimethylurea. In a typical experiment, a 10 mL round-bottom flask was charged with 0.22 g (1.16 mmol) of Et$_3$SiOCH$_2$NMe$_2$ and 0.10 g (0.1.16 mmol) of 1,3-dimethyurea in 5 mL of dichloromethane. The reaction mixture was stirred overnight at room temperature. The solvent and triethylsilanol were removed under vacuum overnight to isolate 1-[(dimethylamino)-methyl]-1,2-dimethylurea MeNH(C=O)NMeCH$_2$NMe$_2$ as a colorless oil, yield 80% The material is thermally stable and does not undergo significant decomposition even on heating at 45°C for 2 days. ^{1}H NMR spectrum, δ, ppm: 2.13 s (6H, CH$_3$), 2.69 d (3H, CH$_3$, *J* 4.8 Hz), 2.87 s (3H, CH$_3$), 3.51 s (2H, CH$_2$), 6.31 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 26.8 (Me), 35.6 (Me), 41.5 (N-Me$_2$), 73.9 (CH$_2$), 160.6 (C=O).

A similar reaction between Et$_3$SiOCH$_2$NMe$_2$ and 1,3-diphenylurea in different molar ratios in dichloromethane failed to yield any dimethylamino-substituted product.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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